भारतीय मानक
*Indian Standard*

**IS 3565 : 2018**

# बोतल से दूध पिलाने के लिए निपल — विशिष्टि

*( पहला पुनरीक्षण )*

# Teats for Feeding Bottles — Specification

*( First Revision )*

ICS 97.190



भारतीय मानक ब्यूरो
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**Price Group 9**

Rubber and Rubber Products Sectional Committee, PCD 13

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber and Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

It is not uncommon to find that teats often contain harmful ingredients which are likely to go into the solution and contaminate the milk or liquid baby food. Further, they often impart undesirable odour, taste or discolouration and also cause irritation to the feeding baby, becoming hard at times. Some of the rubber ingredients are also harmful to the baby and may cause, in acute cases, contact dermatitis.

Taking into consideration the human aspect involved, the Committee had formulate a standard on the subject in order to make the teats harmless and aggregable to the baby. The Committee, while formulating standard, took due note of the lack of facilities for conducting certain biological tests on rubber teats. In the absence of such tests, it is hoped that the accelerators recommended in the standard are strictly adhered to during the manufacture of teats. However, other accelerators, if guaranteed against contact dermatitis and harmful contamination, could be used in the manufacture of teats. The Committee was also in favour of prescribing shapes and sizes for the teats but decided to leave it to the purchaser and the supplier in the absence of standard dimensions for necks of feeding bottles.

This standard was first published in 1966 covering requirements for rubber teats only. Since use of silicone teats has increased over the time period, the Committee responsible for formulation of this standard decided to revise the standard to include requirements for silicone teats also.

Hence in this revision, requirement for both rubber and silicone teats(*see* Annex M) have been stipulated. In order to regulate release of harmful ingredients such as *N*-Nitrosamines and *N*-Nitrosatable substances, vulcanized 2-mercaptobenzothiazole (MBT), anti-oxidants, volatile compounds content, migration of certain elements from natural rubber/silicone teats; requirement limits along with test procedures have been prescribed in this standard.

The Committee considered the possibility of standardizing both the sizes of feeding teats and ranges of flow rates, since many combinations of container systems are in use, it is recommended that all container and feeding teat combinations used are matched components as standardization may not be possible for all sizes and flow rates. The provision of meaningful flow rate information was found difficult because of several factors including hole diameter, teat thickness, hole shape, type of feed, and also how individual infants suck the teat. Accordingly, it was decided not to include a test for flow rate but to recommend that manufacturers provide information on flow rate and hole size that is appropriate to their particular product.

In this revision of standard, considerable assistance has been derived from the following:

a) EN 14350-1:2004 ‘Child use and care articles — Drinking equipment — Part 1: General and Mechanical requirements and tests’,
b) EN 14350-2:2004 ‘Child use and care articles — Drinking equipment — Part 2: Chemical requirements and tests’ ,
c) EN 12868 ‘Child use and care articles- Methods for determining the release of *N*-Nitrosamine and *N*-Nitrosatable substances from elastomer or rubber teats and soothers’ and
d) EN 1400 : 2013 + A1 : 2014 Child use and care articles- Soothers for babies and young children — Safety requirements and test method
e) 93/ 11/EEC Directives
f) US FDA 177.2600

This standard does not purport to address all the safety problems associated with the use of teats. It is the

(*Continued on third cover*)

# *Indian Standard*

# TEATS FOR FEEDING BOTTLES — SPECIFICATION

## *( First Revision )*

### 1 SCOPE

**1.1** This standard prescribes the requirements for reusable teats for feeding bottle made out of natural rubber or silicone rubber.

**1.2** Teats made out of any form of poly vinyl chloride (PVC) and thermoplastic elastomers (TPE) are not part of this standard.

### 2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on the standard may apply the most recent editions of the standards indicated below:

| *IS No.* | *Title* |
|---|---|
| 1070 :1992 | Reagent grade water — Specification (*third revision*) |
| 1699:1995 | Methods of sampling and test for food colours |
| 3025 | Methods of sampling and test (physical and chemical) for water and wastewater |
| (Part 37) : 1988 | Arsenic (*first revision*) |
| (Part 41) : 1992 | Cadmium (*first revision*) |
| (Part 47) : 1994 | Lead (*first revision*) |
| (Part 48) : 1994 | Mercury (*first revision*) |
| (Part 52) : 2003 | Chromium (*first revision*) |
| (Part 56) : 2003 | Selenium (*first revision*) |
| 4905 : 2015/ISO 24153 : 2009 | Random Sampling and Randomization Procedures (*first revision*) |
| 7503 (Part 3) : 1988 | Glossary of terms used in the rubber industry: Part 3 Definitions related to properties and testing. (*first revision*) |
| 9873 (Part 3) : 1999 | Safety requirements for toys: Part 3 Migration of certain elements (*first revision*) |
| 15303 : 2003 | Determination of Antimony, Iron and Selenium in Water by Electro-thermal Atomic Absorption Spectrometric Method |

### 3 TERMINOLOGY

For the purpose of this standard following definitions shall apply in addition to IS 7503(Part 3).

**3.1 Feeding Teat** — Substitute mother's nipple that when attached to a container permits a child to obtain fluid by sucking.

**3.2 Acetone Extract** — Material extracted from teat by acetone under specified conditions.

**3.3 Water Extract** — Material extracted from teat with water under specified conditions.

**3.4 Extractable Protein** — Protein content extracted from latex articles into aqueous phase or into artificial saliva under specified conditions.

**3.5 N- Nitrosamines** — Substance characterized by the =N–N=O functional group, usually formed by the reaction of an amine (primarily a secondary amine) with a nitrosating agent, for example, nitrite, at acidic *p*H.

**3.6 N-Nitrosatable Substance** — Substance which when released into the test solution undergoes nitrosation to form a N-Nitrosamine under specified conditions.

**3.7 Artificial** — To obtain artificial saliva, dissolve 4.2g of sodium bicarbonate, 0.5g of sodium chloride, 0.2g of potassium carbonate and 30.0mg of sodium nitrite in one litre of distilled water. The solution must have a *p*H value of 9.0.

**3.8 Reusable** — Component intended to be used again after first use.

### 4 TYPES

This standard prescribes following two types of teats:

a) *Type 1* — Teats made out of natural rubber, and

b) *Type 2* — Teats made out of silicone rubber.

### 5 REQUIREMENTS

#### 5.1 Material

**5.1.1** The teats shall be made out of natural rubber or silicone rubber, together with necessary compounding and vulcanizing ingredients. In case of natural rubber, solid rubber or latex may be used.

**5.1.2** Teats shall be free from grits, reclaimed rubber or vulcanized waste. The rubber/silicone mix shall not include any ingredient known to be injurious or poisonous to human beings.

**5.1.3** All ingredients used in the manufacture of teats shall be free from harmful substances liable to extraction by contact with milk/liquid food or which may cause development of undesirable odour, taste or discolouration. Softeners, organic accelerators, vulcanizing agents and antioxidants, if incorporated shall not impart any undesirable odour or taste to the finished teats.

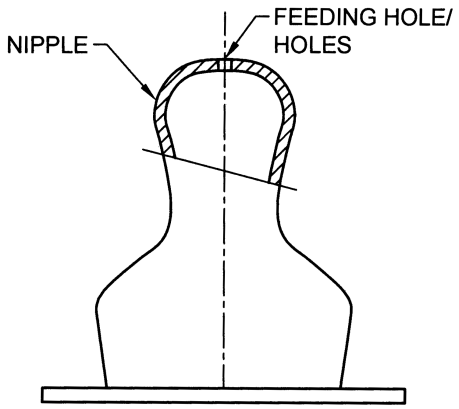
### 5.2 Physical Requirements

**5.2.1** *Workmanship and Finish*

The teats shall be transparent or translucent and shall be free from patches, blisters, porosity, embedded foreign matter and physical defects when examined visually.

**5.2.2** *Shape and Size*

The teat shall have a flat circular bottom, so that it can be firmly attached to a feeding bottle without any leakage (*see* Fig. 1). Besides circular, teats of any other shape or size shall be agreed to between the purchaser and the supplier.




Key

1. Nipple
2. Feeding Hole/Holes

FIG. 1 DESIGN FEATURES OF A FEEDING TEAST

**5.2.3** *Resistance to Autoclaving*

The teat shall show no visual deformation or damage when tested in accordance with Annex A.

### 5.3 Chemical Requirements

**5.3.1** Teats shall also comply with the requirements given in Table 1.

**5.3.2** *Requirements to Check Release of Harmful Ingredients*

The vulcanizing agents 2-mercaptobenzothiazole (MBT), and antioxidants mentioned in Table 2 below do not represent a definitive list.

NOTE — Chemicals other than those mentioned in Table 2 may be used where toxicological evidence, either on the original chemical or any reaction product, is available to demonstrate that no unacceptable risk will be posed when they are used in teats and an appropriate analytical test procedure for determining migration levels exists.

**5.3.3** *Migration of Heavy Metals*

**5.3.3.1** The migration of elements from rubber teats shall comply to the limits given in Table 3.

**5.3.3.2** *Procedure*

Take a test portion of 1000mg of the teat and add 50 times its mass of an aqueous HCl solution at 37 ± 2°C of c(HCl) = 0.07±0.005 mol/l, avoiding heating of the material. Shake for 1 minute. Check the acidity of the mixture. If the *p*H is greater than 1.5, add drop wise, while shaking the mixture an aqueous solution of c(HCl) approximately 2 mol/l [2.0 ± 0.2 mol/l] until the *p*H of the mixture is between 1.0 and 1.5. Protect the mixture from light. Agitate the mixture continuously at 37 ± 2°C for 1 h and then allow standing for 1 h at 37 ± 2°C. Without delay, efficiently separate the solids from the solution, by filtration. If the resulting solution are to be stored for more than one working day prior to elemental analysis, stabilize them by addition of hydrochloric acid so that concentration of stored

**Table 1 Chemical Requirements of Teats**
(*Clause* 5.3.1)

| Sl No. | Characteristic | Requirements | | Method of Test, Ref to Annex |
|---|---|---|---|---|
| | | Type 1 | Type 2 | |
| (1) | (2) | (3) | (4) | (5) |
| i) | Water extract: | | | |
| | a) *p*H | 7 ± 0.5 | 7 ± 0.5 | B |
| | b) Colour | Colourless | Colourless | |
| | c) Turbidity | Not turbid | Not turbid | |
| | d) odour | Odourless | Odourless | |
| ii) | Acetone extract, percent: | | | C |
| | a) Acetone extracted material, percent by mass, *Max* | 3.0 | 3.0 | |
| | b) Free sulphur, percent by mass, *Max* | 0.2 | NA | |
| iii) | Ash content, percent by mass, *Max* | 2.0 | NA | D |
| iv) | Volatile components, percent *Max* | 0.3 | 0.5 | E |
| v) | Extractable protein content, ppm, *Max* | 50 | NA | F |

**Table 2 Tests to Be Carried Out on Materials**
(*Clause* 5.3.2)

| Sl No. | Materials | Migration of Certain Elements (*see* 5.3.3) | N- Nitrosamines and N-Nitrosatables Release (*see* Annex G) | MBT Release (*see* Annex H) | Anti-oxidants Release (*see* Annex J) |
|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (5) | (6) |
| i) | Vulcanised rubber | × | × | × | × |
| ii) | Silicone rubber | × | × | – | – |

solution is approximately c(HCl) = 1 mol/l [*see* IS 9873 (Part 3)].

**5.3.4** *N-Nitrosoamines and N-Nitrosatables*

When tested in accordance with Annex G, the total N-Nitrosoamines and N-Nitrosatables release of any elastomer or rubber component along with tolerance limits shall be as given in Table 4.

**5.3.5** *Determination of MBT*

When elastomeric component of rubber teats tested in accordance with Annex H, the migration of the 2-mercaptobenzothiazole (MBT, CAS No. 149-30-4) release shall not exceed 8 mg/kg (8 ppm).

**5.3.6** *Determination of Antioxidants*

When elastomeric components of rubber teats are tested as given in Annex H, the migration of the antioxidant [2,6-bis (1,1-dimethylethyl)-4-methyl-phenol (BHT], CAS No. 128-37-O) chemical shall not exceed 30 µg/ 100 ml or 60 µg/dm$^2$.

When elastomeric components of rubber teats are tested as given in Annex H, the migration of the antioxidant 2,2'-methylenebis [6-(1,1-dimethylethyl)-4-methyl-phenol) (Antioxidant 2246], (CAS No. 119-47-1) shall not exceed 15 µg/100 ml or 30 µg/dm$^2$. Information regarding suitable HPLC apparatus is given at Annex J).

**5.4 Mechanical Test**

**5.4.1** *Preparation of Samples*

**5.4.1.1** *Re-usable*

Vulcanized rubber (but not silicone products) taken directly from the manufacturer prior to being placed on the market, shall be artificially aged for seven days in an aerated drying cabinet at a temperature of 70 ± 2°C and relative humidity of 65 ± 5 percent. All samples shall be totally immersed in boiling water conforming to IS 1070, Grade 3, for 10 min without touching the walls of the container and then conditioned in accordance with **5.4.1.3**.

NOTE — This procedure is designed to remove any surface coating remaining from manufacturing processes and to ensure that the construction and materials used are stable in boiling water. New samples, preferably from the same batch, shall be used for each test.

**5.4.1.2** *Single-use*

All samples shall be conditioned in accordance with **5.4.1.3**. New samples, preferably from the same batch, shall be used for each test.

**5.4.1.3** *Conditioning*

All samples shall be conditioned for at least 40 h, in a standard atmosphere at a temperature of 27 ± 2 °C and relative humidity of 65 ± 5 percent. Samples shall remain in the conditioning atmosphere until just before the test is carried out. The tests may be carried out in a non-conditioned room.

**5.4.2** *Tear Resistance Test*

The sample of teat should not get puncture when tested

**Table 3 Permissible Levels of Heavy Metals in Teats**
(*Clause* 5.3.3)

| Sl No. | Heavy Metals | Maximum Limit, ppm | | Ref to, IS No. |
|---|---|---|---|---|
| | | Type 1 | Type 2 | |
| (1) | (2) | (3) | (4) | (5) |
| i) | Antimony | 15 | 15 | IS 15303 |
| ii) | Arsenic | 10 | 10 | IS 3025 (Part 37) |
| iii) | Chromium | 10 | 10 | IS 3025 (Part 52) |
| iv) | Mercury | 10 | 10 | IS 3025 (Part 48) |
| v) | Cadmium | 20 | 20 | IS 3025 (Part 41) |
| vi) | Lead | 25 | 25 | IS 3025 (Part 47) |
| vii) | Barium | 100 | 100 | IS 1699 |
| viii) | Selenium | 100 | 100 | IS 15303/IS 3025 (Part 56) |

**Table 4 Permissible Level of N-Nitrosamines and N-Nitrosatables in Teats**
(*Clause* 5.3.4)

| Sl No. | Substance | Maximum Limit mg/kg | Tolerance mg/kg |
|---|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | N-Nitrosamines | 0.01 | 0.01 |
| ii) | N-Nitrosatables | 0.1 | 0.1 |

as per procedure given in Annex K. In case, the teat punctures, another piece should be tested for tensile test.

**5.4.3** *Tensile Test*

This test should be conducted only if the teat punctures in tear resistance test. The sample shall be taken to have passed the test, if no teat tears on tensile test conducted as per test conducted in Annex L.

## 6 PACKING AND MARKING

### 6.1 Packing

Each teat shall be packed in polythene and further packed as agreed to between the manufacturer and buyer and shall include clear legible instructions for the use and hygienic care of the product.

### 6.2 Marking

The following information shall be visible on the packaging or on a leaflet placed inside the product and/or on the external packaging material.

a) Manufacturer's name or trade-mark or the company responsible for placing the product in market or other means of identification, and the address of the manufacturer, distributor or retailer. The particulars may be abbreviated provided that the abbreviation enables the manufacturer, the distributor or the retailer to be identified and easily contacted;
b) Type;
c) Batch number and month and year of production;
d) Number of teats in each package;
e) Instructions for use given in **6.3**, or if these are included in a leaflet within the packaging, a note indicating that this is the case;
f) For products containing natural rubber latex the following information shall be given: 'Produced from natural rubber latex; may cause allergic reactions in some cases'.
g) An indication of the container for which these teats are suitable shall be provided;

NOTE — Additional information on flow rate, hole size or type of use of the feeding teats should be given.

**6.2.1** *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

**6.2.1.1** The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

### 6.3 Instructions for Use

The following information shall be provided:

a) Information for the safe use of the product;
b) Unsuitable common methods of heating which might damage the teats, the following additional instructions shall be provided:
   1) At least one method of cleaning;
   2) Before first use, clean the product;
   3) Unsuitable common methods of cleaning, storage and use which might damage the product.
c) Warnings such as:
   1) Always use this product with adult supervision.
   2) Never use feeding teats as a soother.
   3) Continuous and prolonged sucking of fluids will cause tooth decay.
   4) Always check food temperature before feeding.
   5) For products made out of natural rubber latex shall display, 'Produced from natural rubber latex, may cause allergic reactions in some cases'.

      NOTE — It is recommended that more information relating to possible allergic reactions should be given.

   6) Inspect before each use and pull the feeding teat in all directions. Throw away at the first signs of damage or weakness.
   7) Do not leave a feeding teat in direct sunlight or heat, or leave in disinfectant

('sterilizing solution') for longer than recommended, as this may weaken the teat

8) Before first use place in boiling water for 5 min. This is to ensure hygiene.
9) Clean before each use.
10) Take extra care when microwave heating. Always stir heated food to ensure even heat distribution and test the temperature before serving.

## 7 SAMPLING

Representative samples of the material shall be drawn (*see* IS 4905) and their conformity to this standard shall be determined in accordance with method prescribed in Annex M.

## 8 TEST METHODS

Test shall be conducted following procedures described in Annex A to Annex M.

## 9 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis

# ANNEX A

(*Clause* 5.2.3)

## CHANGE IN PHYSICAL PROPERTIES ON AUTOCLAVING

### A-1 OUTLINE OF THE METHOD

Teats are autoclaved for a fixed time at constant temperature and the change in physical appearance of teats, as examined visually, is reported.

### A-2 APPARATUS

**A-2.1 Autoclave,** capable of being maintained at 121 ± 2 °C and at 0.1 MPa.

**A-2.2 Hot Air Oven**,capable of being maintained at 105 ± 2°C.

### A-3 PROCEDURE

Take three teats and autoclave them in 250 ml of water for 1 h at 121 ± 2°C and 0.1 MPa. Then, keep the teats in a hot air oven maintained at 105 ± 2°C for 1h, and examine the teats after cooling to room temperature for any sign of deterioration such as tackiness, hardness, cracks and discolouration.

# ANNEX B

[*Table* 1, *Sl No*. (i) ]

## METHOD OF TEST FOR *p*H VALUE, COLOUR, ODOUR AND TURBIDITY OF WATER EXTRACT

### B-1 OUTLINE OF THE METHOD

From the aqueous extract, non-aqueous layer, if any, present is separated. *p*H of water extract is determined using a direct reading *p*H meter.

### B-2 REAGENTS

**B-2.1 Buffer Solution**, *p*H 7.0.

### B-3 APPARTAUS

**B-3.1 Beaker***,* 500 ml.

**B-3.2 Beaker**, 100 ml.

**B-3.3 Watch Glass,** of suitable diameter to cover the 500 ml beaker.

**B-3.4 Separating Funnel,** 500 ml.

**B-3.5 *p*H Meter**, equipped with glass electrode and calomel electrode capable of directly reading *p*H, with an accuracy of ± 0.05 *p*H.

**B-4 PROCEDURE**

**B-4.1 Preparation and Purification of Water Extract**

Boil 5 numbers of teats in 300 ml distilled water for 15 min in a 500 ml beaker without touching the walls of the beaker, and covered with a watch glass and allow the mixture to cool to room temperature. Boiling and cooling may be done in an atmosphere free of any gases that may change the *p*H of the aqueous extract. Visually examine the water extract for any change in colour, odour or turbidity.

The extract is transferred to a separating funnel and allowed to stand for 10 min. Non-aqueous layer, if any present is removed. About 75 ml of the aqueous layer is transferred to a clean 100 ml beaker for measuring *p*H.

**B-4.2 Standardization of *p*H Meter**

*p*H meter is standardized using standard buffer solution by following the instructions of the equipment manufacturer.

**B-4.3 Measurement of *p*H**

Introduce the clean electrode in to the aqueous extract taken a 100 ml beaker and measure its *p*H. Keep the equipment in the standby mode and again switch on the instrument to read the *p*H of the extract.

**B-4.4 Report**

The report shall include the two individual values and their average.

# ANNEX C

[*Table* 1, *Sl No*. (ii) ]

**DETERMINATION OF ACETONE EXTRACTABLE MATTER FROM TEATS**

**C-1 OUTLINE OF THE METHOD**

From an unvulcanized rubber mix or vulcanized rubber products, rubber compounding ingredients like process oils, antioxidants, softening agents, free sulphur, etc, if present, can be extracted with low boling organic solvents like acetone. Properties of rubber mix/articles can be altered by varying the quantity of any one or more of the above components.

For the determination of acetone extractable matter, a known weight of sample is repeatedly extracted with acetone in a suitable extraction apparatus. The extract on evaporation leaves behind the extracted matter. This is weighed and percentage of acetone soluble matter is calculated.

**C-1.1 Apparatus**

**C-1.1.1** *Weighing balance*, to weigh accurately to 0.1 mg.

**C-1.1.2** *Soxhlet Extraction Assembly*, attached round bottom flask shall be of capacity about 50 $cm^3$ above that of the extraction cup. Typically extraction cup may be 50 $cm^3$ and round bottom flask be 100 $cm^3$ in capacity.

**C-1.1.3** *Water Bath*, with temperature control of ± 2°C.

**C-1.1.4** *Hot Air Oven*, capable of being maintained at 70 ± 5°C.

**C-1.1.5** *Desicator*, capacity suitable for keeping the round bottom flask of soxhlet extraction assembly.

**C-1.2 Procedure**

Clean the round bottom flask attached to the soxhlet extraction assembly and dry it at 70 ± 5°C for 2h. Cool the flask in desiccator and weigh accurately correct to 0.1 mg.

Weigh accurately about 2 g of the specimen and cut into small pieces. If the specimen is in the form of a sheet, cut it to strips of about 3 mm width. Place the sample on a filter paper and roll into a cylinder. Ensure that the sample pieces do not touch each other (The diameter of sample cylinder is such that it can be placed in the extraction cup). The length of the sample cylinder shall be atleast 10 mm lower than the maximum level of acetone in the extraction cup.

Place the sample in the extraction cup. Take about

60-70 ml. acetone in the round bottom flask. Set up the soxhlet extraction assembly and heat on a water bath. The rate of heating may be such that one extraction takes place in every 3-4 min. Extract the specimen continuously for 16h.

After extraction detach the round bottom flask and evaporate off the acetone over the water bath without boiling. Remove the flask from the water bath just prior to the disappearance of the last traces of solvent. Allow the remaining solvent to evaporate in air. Dry the flask at 70 ± 5 °C for 2 h in an electric oven. Cool the flask in a desiccator and weigh.

**C-1.3 Calculation**

$$\text{Acetone extractable matter (percent)} = \frac{M_3 - M_2}{M_1} \times 100$$

where

$M_1$ = mass of the teat sample,

$M_2$ = mass of empty round bottom flask, and

$M_3$ = mass of the round bottom flask with acetone extracted matter.

## C-2 DETERMINATION OF FREE SULPHUR FROM ACETONE EXTRACTABLE MATTER IN RUBBER TEATS

Free sulphur in the acetone extract is oxidised to sulphate, which is precipitated as barium sulphate and estimated gravimetrically.

**C-2.1 Reagents**

**C-2.1.1** *Bromine Water* — Saturated aqueous solution.

**C-2.1.2** *Nitric Acid* — Specific gravity 1.50.

**C-2.1.3** *Zinc Oxide-Nitric Acid Solution* — Add 200g zinc oxide to 1 $dm^3$ of nitric acid (sp. gravity 1.42).

**C-2.1.4** *Potassium Chlorate,* Crystals.

**C-2.1.5** *Picric Acid* — Saturated aqueous solution.

**C-2.1.6** *Barium Chloride*, aqueous solution, 100 g/litre.

**C-2.1.7** *Hydrochloric Acid*, specific gravity 1.18.

**C-2.1.8** *Silver Nitrate* — 1 percent aqueous solution.

**C-2.2 Apparatus**

**C-2.2.1** *Water Bath*

**C-2.2.2** *Hot Plate*

**C-2.2.3** *Silica Crucible*, with lid, capacity 80 ml.

**C-2.2.4** *Muffle Furnace*, capable of heating up to 1 000°C, having temperature sensing device accurate to 25°C.

**C-2.2.5** *Desiccator*

**C-2.3 Procedure**

To the flask containing acetone extracted matter add 10 ml of zinc oxide nitric acid solution and 2 to 3 ml of bromine water and cover with a watch glass. Allow to stand for 30 min and then heat on a water bath to foamy syrup. Add 10 ml. of nitric acid and then heat on a hot plate until all bromine is expelled. If the residue in the round bottom flask shows the presence of organic matter or carbon, add a few millilitres of nitric acid and a few crystals of potassium chlorate and evaporate at boiling. Repeat this evaporation until all carbon is removed and the solution is clear, colourless or light yellow.

Evaporate the mixture on a hot plate, cool and add 10ml hydrochloric acid and evaporate to dryness, avoiding spattering. Repeat additions of acid and evaporation until oxides of nitrogen are not evolved.

Cool the flask, add 50 ml. dilute hydrochloric acid (1:6) and digest the contents on a hot plate until dissulation of the contents is as complete as possible. Filter this solution while hot through a Whatman No. 1 filter paper and collect the filtrate to a 500 ml beaker. Wash the residue on the filter paper 3-4 times with distilled water and the washings are also collected in to the filtrate in the 500 ml beaker. The combined volume of filtrate may be about 300 ml. Add 10 ml saturated picric acid solution. From a burette add barium chloride solution dropwise while stirring vigorously to precipitate sulphate as barium sulphate. Cover the beaker with a watch glass and digest the precipitate overnight at 60°-80°C. Filter the barium sulphate through an ash less filter paper (Whatman No. 42 or 542). Transfer completely the precipitate to the filter paper and wash it repeatedly with water, until the washing is free of chloride (Filtrate shall not give a white precipitate with silver nitrate which is soluble in ammonia solution.). Place the precipitate in the filter paper in a clean silica crucible which has been dried and weighed. Dry, incinerate and finally ignite the precipitate at 650-900°C in a muffle furnace to constant weight. Cool in a desicator and weigh.

**C-2.4 Calculation**

Calculate the sulphur content as follows:

$$\text{Sulphur content, (percent m/m)} = 0.137\ 37 \times \frac{(M_3 - M_2)}{M_1} \times 100$$

where

$M_1$ = mass of teat sample,

$M_2$ = mass of the empty crucible, and

$M_3$ = mass of crucible and barium sulphate.

# ANNEX D

[*Table* 1, *Sl No*. (iii)]

## DETERMINATION OF ASH

### D-1 APPARATUS

**D-1.1 Platinum or Silica Dish**, having a capacity of 100 ml.

**D-1.2 Muffle Furnace**, capable of heating up to 1 000 °C and having temperature sensing device accurate to ± 25 °C.

**D-1.3 Bunsen Burner**

**D-1.4 Desiccator**

### D-2 PROCEDURE

Heat the platinum dish to redness, cool to room temperature in a desiccator and weigh. Take about 5.0 g of the material in a watch glass and weigh accurately. Transfer about three quarters of this quantity to the platinum dish and heat on a Bunsen burner so that the material burns gently at the surface. When about half of the material is burnt away, stop heating, cool and add the remainder of the material. Weigh the watch glass again and find, by difference, the exact mass of sample transferred to the platinum dish. Heat again as before till the material is completely charred. Incinerate in a muffle furnace at 550 °C to 650 °C for 1 h. Cool to room temperature in a desiccator and weigh. Repeat incineration, cooling and weighing until the difference between two successive weighing is less than 1 mg.

### D-3 CALCULATION

$$\text{Ash, percent by mass} = \frac{(M_4 - M_1)}{(M_2 - M_3)} \times 100$$

where

$M_1$ = mass of empty dish,

$M_2$ = mass of watch glass and sample,

$M_3$ = mass of dish and ash, and

$M_4$ = mass of crucible and ash.

# ANNEX E

[*Table* 1, *Sl No*. (iv) ]

## DETERMINATION OF TOTAL VOLATILE MATTER

### E-1 APPARATUS

**E-1.1 Weighing Balance**, with accuracy of 1 mg.

**E-1.2 Hot Air Oven**, which can be heated to at least 110 °C and having an accuracy of ± 2 °C.

**E-1.3 Desiccator**

**E-1.4 Suitable Dish to Hold the Sample**

### E-2 PROCEDURE

In a suitable dish, which has been previously dried and weighed, weigh accurately about 10 g of the material. Heat the dish with material for 6 hours in an oven maintained at 105 ± 2 °C. Cool the dish along with the material in a desiccator and weigh. Heat the dish with material again for 30 min and weigh. Repeat the process until the loss in mass between two successive weighing is less than 1 mg. Record the constant mass obtained.

### E-3 CALCULATION

$$\text{Total volatile matter percent by mass} = \frac{(M_1 - M_2)}{(M_1 - M_3)}$$

where

$M_1$ = mass of the dish with the material before heating, in g;

$M_2$ = mass in g of the dish with the material after heating, in g; and

$M_3$ = mass of the empty dish, in g.

# ANNEX F

[*Table* 1, *Sl No*. (v) ]

## ESTIMATION OF EXTRACTABLE PROTEIN

### F-1 PRINCIPLE

Extractable proteins are extracted from teats by water. Protein in the extract is concentrated and estimated colourimetrically using Phenol Folin Reagent.

### F-2 APPARATUS

**F-2.1 Standard Laboratory Glasswares**

**F-2.2 Centrifuge**, with 15ml capacity tubes and minimum speed of 3 000 rpm.

**F-2.3 Centrifuge,** with 50ml capacity tubes and a minimum speed of 3 000 rpm.

**F-2.4 Vortex Mixture**

**F-2.5 Spectrophotometer**, for measurement of colour at 750 nm.

### F-3 REAGENTS

**F-3.1 Trichloro Acetic Acid** — 35 percent *w/v* aqueous solution.

**F-3.2 Phospho Tungstic Acid** — 40 percent *w/v* aqueous solution.

**F-3.3 Sodium Hydroxide**, 0.1 M.

**F-3.4 Sodium Hydroxide**, 0.25 M.

**F-3.5 Reagent A,** 6 percent sodium carbonate in 0.2M sodium hydroxide.

**F-3.6 Reagent B**, 1.5 percent copper sulphate in 3 percent sodium citrate.

**F-3.7 Reagent C**, 50 ml reagent A mixed with 1 ml reagent B.

NOTE — Reagent C may be prepared at the time of use.

**F-3.8 Folin Reagent** — 3 Parts of phenol folin and 1 part of water.

**F-3.9 Bovine Serum Albumin**

### F-4 PROCEDURE

**F-4.1 Elution of Proteins**

Weigh accurately about 0.3-0.5g of teat portion and cut it to about 0.5 mm size. Leach the sample in 30 ml of distilled water for 4h at ambient temperature. Stir the water at intervals of 30 min. Filter the solution through Whatman No.1 (or equivalent) filter paper.

**F-4.2 Protein Purification and Concentration**

To 6 ml extract (obtained in **F-4.1**) contained in a 15 ml centrifuge tube, add 1 ml trichloro acetic acid solution, mix well using a vortex mixer, and allow to stand for 5 min. To this mixture add 1 ml of phospho tungstic acid solution and mix well. The proteins are precipitated and allowed to stand for 20 min. Centrifuge the mixture for 30 min; keep the tube inverted to remove the liquid fraction. Dissolve the precipitate in 0.8 ml of 0.1M sodium hydroxide solution.

**F-4.3 Dissolution of Precipitated Protein**

Re-dissolve the precipitate obtained in **F-4.2** (which is very thin, if protein concentration is low) in 0.8 ml 0.25 M sodium hydroxide solution for at least 20 min.

NOTE — In the event that the precipitate is abundant, the volume of sodium hydroxide may be increased, so that the photometric reading does not fall outside the calibration curve. In such case, pipette out 0.8 ml protein solution and proceed to **F-4-4**.

**F-4.4 Protein Estimation.**

To the above solution add 0.1 ml of folin reagent, mix well using a vortex mixture, keep for 30 min at room temperature and read the absorbance at 750 nm against a blank.

**F-4.5 Preparation of Calibration Curve.**

Bovine serum albumin is used as reference protein. 0.25 g of this protein is dissolved in water and dilute to 250 ml in an volumetric flask to get 1 000 ppm solution. 10 ml of this solution is diluted to 100 ml to get 100 ppm solution.

Standard curve is plotted in the appropriate concentration range 0-60 µg/ml. 6 ml of each standard solution is taken through the procedure described in **F-4.2** and **F-4.4**.

### F-5 CALCULATION

Concentration of protein in the extract is read from the calibration curve.

Extractable protein content in the sample = $\frac{30C}{W}$ µg/g

Where C is the concentration of protein in the sample is µg/ml, as obtained from the calibration curve and w is the mass of the sample, in g.

# ANNEX G

( *Clause* 5.3.4)

## DETERMINATION OF N-NITROSAMINES AND N-NITROSATABLE SUBSTANCES

### G-1 PRINCIPLE

N-Nitrosamines and N-Nitrosatable substances are extracted into a nitrite-containing artificial saliva salt solution. After concentration and, in the case of N-Nitrosatable substances, after conversion, the final test solutions are examined for N-Nitrosamines by gas chromatography (GC) employing a chemiluminescence detector or other suitable validated analytical technique substances. The analysis shall be carried out in an atmosphere free from volatile N-Nitrosamines and N-Nitrosatable substances. The N-Nitrosamine and N-Nitrosatable substances released are expressed as N-Nitrosamines released, in micrograms per kilogram (µg/kg), of the sample.

NOTE — N-Nitrosamines can endanger human health owing to their toxicity.

### G-2 REAGENTS

**G-2.1 Sodium Hydrogen Carbonate**

**G-2.2 Sodium Chloride**

**G-2.3 Potassium Carbonate**

**G-2.4 Sodium Nitrite**

NOTE — Sodium nitrite, on exposure to air or oxygen is easily oxidized to sodium nitrate. Ensure that this chemical undergoes minimum exposure to air or oxygen during storage or handling. Even when properly stored, this chemical shall have a shelf life of only two years.

**G-2.5 Hydrochloric Acid Solution** — 0.1 M.

**G-2.6 Sodium Hydroxide Solution** — 0.1 M.

**G-2.7 Artificial Saliva Salt Solution** — Dissolve 4.2 g of sodium hydrogen carbonate, 0.5 g of sodium chloride, 0.2 g of potassium carbonate and 30 mg of sodium nitrite in water and dilute to 900 ml with water. Adjust to *p*H 9.0, if necessary by adding hydrochloric acid solution or sodium hydroxide solution drop by drop. Transfer into a 1 litre volumetric flask and dilute to the mark with water.

NOTE — This solution can have a shelf-life of not more than two weeks when stored in a stoppered bottle having a minimum air space above the liquid.

**G-2.8 Dichloromethane** — distilled in glass and checked for the absence of nitrosamines and nitrosatable substances.

**G-2.9 Kieselguhr, from Liquid-Liquid Extraction** — (surface 1 $m^2/g$, pore size 3 000 nm to 8 500 nm, particle size 150 µm to 650 µm); heated to 200°C for 1 h, cooled and washed with dichloromethane.

NOTE — An alternative separation material can be employed provided it has been validated against kieselguhr.

**G-2.10 *n*-Hexane**

**G-2.11 Hydrochloric Acid Solution** — 0.1 M.

**G-2.12 Sodium Hydroxide Solution** — 1 M.

**G-2.13 Purified Nitrogen**

**G-2.14 Anti- Bumping Granules**

**G-2.15 Sintered Glass Frits for Columns**

**G-2.16 Acetone**, or other suitable solvent.

**G-2.17 Standard Solutions of N-Nitrosamines** — Prepare a solution(s) in the n-hexane of known amount of the N-Nitrosamines to be determined within the concentration range of 100 ng/ml to 300 ng/ml. Alternatively, certified solutions may be used to achieve the same concentration range.

The following N-Nitrosamines have been identified as of concern in rubber and elastomeric teats and soothers. However, this list is not exhaustive:

a) N-Nitrosodimethylamine (NDMA)
b) N-Nitrosodiethylamine (NDEA)
c) N-Nitrosodipropylamine (NDPA)
d) N-Nitrosodibutylamine (NDBA)
e) N-Nitrosopiperidine (NPIP)
f) N-Nitrosopyrrolidine (NPYR)
g) N-Nitrosomorpholine (NMOR)
h) N-Nitrosodibenzylamine (NDBzA)
j) N-Nitrosodiisonoylamine (NDiNA),that is N-Nitroso3,5,5-trimethylhexylamine
k) N-Nitroso N-methyl N-phenylamine (NMPhA)
m) N-Nitroso N- ethyl N-phenylamine (NEPhA).

Should other N-Nitrosoamines be detected, they should also be determined as described.

**G-2.18** Internal standard solution of N-Nitrodiisopropylamine (NDiPA), free from other N-Nitrosamines, 200 ng/ml in the acetone or other suitable solvent.

NOTE — N-Nitrosamines are degraded by ultra-violet light. Exposure of extracts or standards to sources such as sun-light or fluorescent tube light should be avoided. The samples and standards should be protected by wrapping in aluminium foil and stored in the dark at a temperature of less than 5°C.

**G-2.19 Anhydrous Sodium Sulphate,** (granular) or suitable Whatman phase separating filter.

Prewash 30 g of sodium sulphate with 25 ml of the dichloromethane.

**G-2.20 Ammonia Solution** — 0.1 M.

**G-2.21 Sand, Acid Washed and Calcined**

**G-3 APPARATUS**

**G-3.1 Normal Laboratory Apparatus** — Any glass apparatus washed with acidic cleaning agents shall be treated with the ammonia solution, rinsed with water and dried, prior to use in the tests.

**G-3.2 Oven**, maintained at a temperature of 40 ± 2°C.

**G-3.3 Glass Column**, with outlet and polytetrafluoroethylene (PTFE) stopper; column length approximately 300 mm, internal diameter approximately 26 mm.

**G-3.4 Glass Column with Outlet and PTFE Stopper**,column length approximately 300mm, internal diameter approximately 15 mm.

**G-3.5 Kuderna - Danish (K-D) Evaporative Flask and Concentrator**, modified with a graduated collecting vessel and an air cooler with a floating or expansion sphere.

NOTE — An alternative concentrator can be employed provided it has been validated against the K-D system.

**G-3.6 Water Bath**, capable of maintaining temperatures in the range 40°C to 60°C.

**G-3.7 Ampoules,** welted-edged and capable of being closed with flanged rings and PTFE-coated septa (to ensure that the septa are free from N-Nitrosamines).

**G-3.8 Sealing Tongs for the Ampoules**

**G-3.9 Glass Wool, Washed with the Dichloromethane**

**G-3.10 Separating Funnel**, 200 ml.

**G-3.11 Separating Funnel**, 100 ml.

**G-3.12 Chemiluminescence Detector**, of adequate sensitivity (Thermal Energy Analyzer)

NOTE — An alternative analytical detector can be employed provided it has been validated against TEA.

**G-3.13 Gas Chromatograph** (GC) — The GC system to be used is left to the discretion of the analyst. However, the laboratory shall demonstrate that conditions have been optimized to take account of the following:

a) System(s) shall separate the N-Nitrosamines named in this standard, such that their peak areas can be compared with that due to the internal standard;

b) System shall separate N- Nitrosodimethylamine and N-Nitrosodiethylamine from the named N-Nitrosamines.

NOTE — The following guidance is provided on chromatographic systems which can be suitable to obtain the desired separations. However, conditions will vary between laboratories and each laboratory should ensure that adequate separation is achieved for their chosen system(s). Two columns can be required to separate all *N*-Nitrosamines and to obtain adequate sensitivity for NDBzA.

The following conditions have been found suitable for the determination of volatile *N*-Nitrosamines:

a) *Example 1* : Packed Columns

1) Injector temp : 200 °C.

2) Oven temp : 200 °C.

3) Column : either (2.5-3.0) m glass, external diameter 1/8" packed with:

   i) either 15 percent Carbowax 20M, TPA on Chromsorb WHP 100/120 mesh

   ii) or 10 percent Carbowax 20M, 2 percent KOH on Chromsorb WHP 100/120 mesh.

   iii) or (4.0-5.0) m glass, external diameter 1/8" packed with 15 percent SP 1200, 1 percent $H_3PO_4$ on Chromsorb WAW 100/120 mesh.

4) Pyrolyser temp — 480°C.

5) Carrier gas — Argon, helium or nitrogen at a flow rate of approximately 20 ml/min.

6) Coupling — Either direct between GC column and pyrolysis oven, or using an interface heated at 250°C.

The following modifications have been found suitable for the determination of alkyl phenyl N-Nitrosamines:

1) Injector temp : 150°C.

2) Oven temp : approximately (120 – 130)°C.

3) Column : 2.0 m glass, external diameter ¼", internal diameter 2.0 mm, packed with: either 10 % OV-101 on Chromsorb WHP 80/100 mesh or 3% OV-225 on Chromsorb WHP 80/100 mesh.

4) Pyrolyser temp : 480 °C.

5) Interface temp : 250 °C.

b) *Example 2* **:** Capillary Columns

**Either:**

1) Injector temp : 200 °C.

2) Oven temp : 60 °C, 230 °C

(10 °C/min).

3) Column : 25.0 m fused silica 0.53 mm FFAP 1 µm.
4) Pyrolyser temp : 480 °C.
5) Interface temp : 250°C.

Or

1) Injector temp : 50°C 1 min, 200°C (75°C/ min).
2) Oven temp : 40°C 7min, 60°C (1°C/min ), 230°C (14°C/min).
3) Column : 30.0 m fused silica 0.53 mm SE-54 film 2 µm.
4) Pyrolyser temp : 480°C.
5) Interface temp : 250°C.

## G-4 PROCEDURE

### G-4.1 Migration from Teats

**G-4.1.1** First remove any non-rubber or elastomeric components from the teats. Weigh a minimum of 10 g of teats per individual analysis that is about 40 g. Transfer to a beaker of boiling distilled water and boil for 10 min using the minimum quantity of water necessary to cover the teats. Remove the teats from the water with tweezers or tongs. Allow to cool to room temperature and then cut each longitudinally into two parts and air dry.

**G-4.1.2** Weigh a minimum of 10 g of the prepared teats to the nearest 0.1 g and place into a 50 ml conical flask. Transfer, by pipette, 40.0 ml of the artificial saliva solution. Close with a ground glass stopper and gently shake to ensure the teats are covered by the solution, and stand the closed flask at 40 ±2 °C for 24 h (±30 min) in the oven.

NOTE — If the mass of the teats taken is greater than 10g, the reagents and containers used should be increased proportionally throughout the analysis, except for the volume of the internal standard, which will remain at 1.0 ml.

**G-4.1.3** Decant the solution from the flask into a 50 ml measuring cylinder, closed with a ground glass stopper. Wash the teats with 4.0 ml of the artificial saliva solution and add the washings to the measuring cylinder. Dilute to 50.0 ml with distilled water and mix.

**G-4.1.4** Transfer by pipette 10.0 ml of this solution into a 25 ml conical flask and close with a ground glass stopper. This is Solution B.

**G-4.1.5** The remaining solution (40.0 ml) is Solution A**.**

## G-5 ISOLATION OF N-NITROSAMINES IN SOLUTION A

Transfer by pipette 1.0 ml of the internal standard solution (**G-2.18**) and 1.0 ml of the sodium hydroxide solution (*see* **G-2.12)** into solution A (*see* **G-4.1.5)** contained in the measuring cylinder.

NOTE — The clean-up procedure for the test solution can be achieved by either Method A or Method B.

### G-5.1 Method A

Add 25 g of the Kieselguhr or suitable separation material to the 26 mm internal diameter glass column closed at the bottom with the glass wool. Cover the top of the column with the sintered glass frit or with an approximately 1 cm thick layer of the sand.

When filling the column, gently tap its outside wall to attain a more uniform packing.

**G-5.1.1** Stopper and shake the measuring cylinder containing the solution and slowly add it to the prepared kieselguhr, or equivalent, column (*see* **G-5.1**).

Distribute the sample as the stationary phase on the porous matrix within 10 min to 15 min. A dry zone, approximately 50 mm to 70 mm wide remains in the lower part of the column.

**G-5.1.2** Pour 60 ml to 80 ml of the dichloromethane on to the column and, within 15 min to 25 min, collect the extract (approximately 40 ml) into the K-D flask, or equivalent, regulating the drip rate with the aid of the PTFE stopper.

NOTE — During elution with dichloromethane, the dry zone shrinks to 15 mm to 30 mm. This process is easily observed because of the different toning of the specimen- dampened and dichloromethane — dampened kieselguhr, or equivalent. It is important for this dry zone capacity not to be exhausted, otherwise the specimen can contain water.

### G-5.2 Method B

Stopper and shake the measuring cylinder containing the solution **(***see* **G-5)** and slowly add it to the separating funnel.

**G-5.2.1** Add a minimum of 20 ml of the dichloromethane and shake vigorously for 1 min. Allow the liquid phases to separate and, if necessary, centrifuge to break up any emulsions. Collect the lower layer and pass it through 30 g of the prewashed sodium sulfate or suitable phase separation filter into the K-D flask, or equivalent.

**G-5.2.2** Repeat the procedure given in **G-5.2.1** twice more.

**G-5.2.3** Wash the sodium sulfate or suitable phase separation filter with 25 ml of the dichloromethane and add it to the K-D flask, or equivalent.

**G-5.2.4** *Concentration of N-Nitrosamines in Solution A*

**G-5.2.4.1** To the K-D flask, or equivalent, containing the extract from Method A or Method B, add 2 ml of

the n-hexane and two or three of the anti-bumping granules. Attach the air cooler to the K-D flask, or equivalent. Concentrate the solution to a volume of 4 ml to 6 ml in the water bath starting at (40 ± 2)°C and slowly raising the temperature to 60 ± 2ºC (approximately 2°C/min ) to avoid sample loss. Allow the solution to cool and rinse the walls of the evaporation and concentration system with approximately 2 ml of the dichloromethane.

**G-5.2.4.2** Remove the air cooler from the K-D flask, or equivalent. Gently pass the nitrogen over the solution in the concentrator to reduce the solution to a volume of approximately 1 ml. Leave to equilibrate to room temperature and then transfer it to the welted–edge ampoule **(** *see* **G-3.7)** and close with the septum and flanged ring.

Regulate the flow of nitrogen to ensure that the depression produced on the surface of the concentrated extract does not exceed 4 mm to 5 mm in depth, otherwise spilling or over-cooling of the extract may occur. It is important that the volumes do not fall below the minimum values quoted in the concentration step because of the volatility of the N-Nitrosamines. If the concentrate is to be kept for longer than 1 h before analysis, store it in the dark at a temperature less than 5 ºC.

**G-5.2.5** *Isolation of N-Nitrosatable Substance as N-Nitrosamines in Solution B*

**G-5.2.5.1** To Solution B ( *see* **G-4.1.4)**, add 1.0 ml of the hydrochloric acid solution **(** *see* **G-2.11)** by pipette and shake to mix (this gives a *p*H solution value of approximately 1.4). Allow to stand in the dark for 30 min.

**G-5.2.5.2** Add 2.0 ml of the sodium hydroxide solution ( *see* **G-2.12)** to make the solution alkaline and 1.0 ml of the internal standard solution by pipette and shake.

NOTE — The clean-up procedure of the test solution can be achieved by either Method C or Method D.

**G-5.3 Method C**

Prepare an 8 g kieselguhr, or suitable separation material, column using the 15 mm internal diameter glass column.

Transfer the solution obtained from **G-5.2.5.2** on to this column.

Pour 25 ml to 30 ml of the dichloromethane on to the column and collect the extract (approximately 15 ml) into the K-D flask, or equivalent, regulating the drip rate with the aid of the PTFE stopper.

**G-5.4 Method D**

**G-5.4.1** Add the solution **G-5.2.5.2** to the separating funnel.

**G-5.4.2** Add a minimum of 10 ml of the dichloromethane and shake vigorously for 1 min . Collect the lower organic layer into the K-D flask, or equivalent, as described in **G-5.2.1**.

**G-5.4.3** Repeat the procedure given in **G-5.4.2** twice more.

**G-5.4.4** Wash the sodium sulfate or phase separation filter and add it to the K-D flask, or equivalent.

**G-6 Concentration of N-Nitrosatable compounds as N-Nitrosamines in Solution B**

Concentrate the extract from Method C or Method D to a final volume of approximately 1ml as described in **G-5.2.4.1** and **G-5.2.4.2**.

**G-6.1 Blank Test**

This is conducted by following all the chosen procedures specified in **G-4.1.2** to **G-5.4.4** without the teats and the migration stage **G-4.1.2**.

**G-6.2 Chromatography**

Inject 1 µl to 10 µl of the extract into the GC/chemiluminescence detector unit under the optimized conditions. Also analyze an equal volume of the standard solution and the internal standard solution.

It is recommended that, to obtain reliable results, the analysis should be carried out on the same day as the preparation of the extract. If this is not possible, store the extracts and standards in the dark at a temperature less than 5 ºC.

**G-7 INTERPRETATION OF RESULTS**

**G-7.1 N-Nitrosamine Content of Solution A**

**G-7.2** Calculate the amounts of each of the individual N-Nitrosamines using following formula.

$$M\,(\mu g.kg) = \frac{5F.A_{NA}}{4\,^{A}NDiPA^{R}} \qquad \ldots(1)$$

where

$M$ = quantity of N-Nitrosamine migrating from the sample into the saliva test solution in µg/kg, corrected with reference to the added $N_{DiPA}$ internal standard recovery rate;

$F$ = factor calculated using Equation 2;

$^{A}_{NA}$ = peak area of the identified N-Nitrosamine migrating from the sample into the saliva test solution (Solution A); and

$^{A}_{NDiPA}{}^{R}$ = peak area of the $N_{DiPA}$ internal standard recovered from test Solution A.

$$F = \frac{V.C.A_{NDiPA^I} \cdot V_{NASTD}}{G.A_{NASTD} \cdot V_{NDiPA^I}} \quad \ldots(2)$$

where

$V$ = volume of added $N_{DiPA}$ internal standard in ml;

$C$ = concentration of the identified N-Nitrosamine in the standard solution in µg/l;

$G$ = weighed portion of sample material in g;

$A_{NASTD}$ = peak area of the identified N-Nitrosamine in the standard solution;

$A_{NDiPA}{}^{I}$ = peak area of the direct injection of $N_{DiPA}$ internal standard;

$V_{NASTD}$ = injected volume of the N-Nitrosamine standard in µl; and

$V_{NDiPA}{}^{I}$ = injected volume of the added $N_{DiPA}$ internal standard, in µl.

**G-7.3** Calculate the total N-Nitrosamine content by adding together the amount of the individual N-Nitrosamines detected. If no measurable instrumental response is observed for an individual N-Nitrosamine, that is 3 time the background noise, it shall be recorded as 'Not Detected' or 'ND' and its value treated as zero.

A product will comply with this standard if the total quantity of N-Nitrosamines detected is less than 0.01 mg/kg of elastomer or rubber, after applying the analytical correction in **G-9**.

## G-8 N-NITROSATABLE CONTENT OF SOLUTION B, CALCULATED AS N-NITROSAMINES

**G-8.1** Calculate the amounts of each of the individual N-Nitrosamines detected using formula 2 and 3. The quantities of individually determined N-Nitrosamines calculated in Solution A shall be subtracted from the values obtained.

$$M\,(\mu g/kg) = \frac{5F.A_{NA}}{A_{NDiPA^R}}$$

where

$M$ = quantity of N-Nitrosamine migrating from sample into the saliva test solution in µg/kg, corrected with reference to the added $N_{DiPA}$ internal standard recovery rate;

$F$ = factor calculated using formula 2;

$A_{NA}$ = peak area of the identified N-Nitrosamine migrating from the sample into the saliva test solution (Solution B); and

$A_{NDiPA}{}^{R}$ = peak area of the $N_{DiPA}$ internal standard recovered from test Solution B.

**G-8.1.1** Calculate the total N-Nitrosatable content by adding together the amounts of the individual N-Nitrosatable detected corrected for the amounts of the individual N-Nitrosamines calculate in solution A. If no measurable instrumental response is observed for an individual N-Nitrosamines that is 3 times the background noise. It shall be recorded as 'Not Detected' or 'ND' and its value treated as zero.

A product will comply with this standard if the total quantity of N-Nitrosatables detected is less than 0.1 mg/kg of elastomer or rubber, after applying the analytical correction in clause **G-9**.

## G-9 ANALYTICAL CORRECTION

**G-9.1** Any analytical result obtained in **8** which are above the limits specified in **G-7.3** and **G-9.2** shall be adjusted by subtracting the analytical correction given below from the result to provide adjusted analytical results.

**G-9.2** Analytical correction for N-Nitrosamines : 0.01 mg/kg

Analytical correction for N-Nitrosatables substances : 1 mg/kg.

NOTE — Due to the precision of the methods specified in this standard, an adjusted analytical correction is required to take into account the inherent variability in measurement shown by the inter-laboratory trials.

**G-9.3** Products are deemed to comply with this standard, if the adjusted analytical result is less than the limits specified in **G-7.3** and **G-8.1.1**.

*Example :*

Analytical result for N-Nitrosamines = 0.018 mg/kg

Analytical correction = 0.01 mg/kg

Adjusted analytical result = 0.018 mg/kg – 0.01 mg/kg
= 0.008 mg/kg.

This would be deemed as complying with this standard (N-Nitrosamines limit, 0.01 mg/kg).

## G-10 CONFIRMATION OF N-NITROSAMINES DETECTED

If the apparent total N-Nitrosamines content determined in the saliva test solution exceeds, or is equal, to the limits specified in **G-7.3** and **G-8.1.1**, the N-Nitrosamines identified and their quantities shall be confirmed in one of the following ways:

a) By placing an aliquot of the remaining test solution in a clear, UV transparent glass vial and subjecting it to UV radiation (3 h, wavelength = 366 nm) along with a similarly prepared standard solution in a separate vial. On GC analysis any peaks due to the presence

of N-Nitrosamines will disappear or be substantially reduced due to decomposition. However, if the sample peak is not substantially reduced after irradiation, the initial peak was a false positive and no further investigation for the presence of N-Nitrosamines is required;

b) By the use of at least one other column with a stationary phase having a different polarity; or

c) By mass spectrometry.

If it is found that some peaks do not correspond to N-Nitrosamines after following the above procedures, re-calculate the total N-Nitrosamines content including only those peaks due to N-Nitrosamines**.**

### G-11 TEST REPORT

**G-11.1** The work carried out by the testing laboratory shall be covered by a report which accurately, clearly and unambiguously presents the test result, preferably, unadjusted and adjusted analytical result for individual and total N-Nitrosamines and N-Nitrosatable substances should be included.

**G-11.2** Each test report shall include at least the following information:

a) Name and address of testing laboratory and location where the test was carried out when different from the address of the testing laboratory;

b) Unique identification of report (such as serial number) and of each page, and total number of pages of the report;

c) Description and identification of the laboratory sample;

d**)** Description of the sampling procedure, where relevant;

e) Date of receipt of the laboratory sample, and date(s) of performance of test; and Identification of the test specification or description of the method procedure.

## ANNEX H

(*Clause* 5.3.5)

### DETERMINATION OF 2-MERCAPTOBENZOTHIAZOLE (MBT) AND ANTIOXIDANTS RELEASE

### H-1 PRINCIPLE

MBT and its metal-salts are determined quantitatively following extraction into aqueous migration liquids. MBT is identified and determined by high performance liquid chromatography (HPLC) and ultra violet (UV) detection at a specific wavelength, either by direct injection of the aqueous migration liquid, or in a concentrated solution. The identification is confirmed by comparing the UV- spectrum of the sample peak produced by a diode array detector with the spectrum of the peak of an authentic MBT- sample.

The method is also used for the qualitative and quantitative determination of the antioxidants 2,6-bis (1,1-dimethylethyl)-4-methyl-phenol (Antioxidant BHT) and 2,2'-methylenebis (6-(1,1-dimethylethyl)-4-methyl-phenol) (Antioxidant 2246). They too are identified and determined by HPLC and UV-detection at a specific wavelength. The identification is confirmed by comparing the UV-spectra of the sample peaks produced by a diode array detector with the spectra of the peaks of authentic substances. For unknown samples a further identification step by thin layer chromatography (TLC) or gas liquid chromatography (GLC) is recommended.

### H-2 APPARATUS

**H-2.1** HPLC with a 20 µl injection loop diode array detector connected to an integrator or personal computer with chromatography software.

**H-2.2** HPLC-column capable of separating MBT from the antioxidants and fully resolving the antioxidants such that the peaks do not overlap by more than 1 percent peak area with each other and with interferences arising from other sample ingredients.

**H-2.3 Reagents — Chemicals (Analytical Reagent Grade Unless Otherwise Specified)**

**H-2.3.1** *Water,* HPLC grade.

**H-2.3.2** *Acetonitrile*, HPLC grade.

**H-2.3.3** *Distilled Water*

**H-2.3.4** *Dichoromethane*, Residue analysis grade.

**H-2.3.5** *Anhydrous Sodium Sulphate*

**H-2.3.6** *Acetic Acid*, 3 percent (*w*/*v*) aqueous solution.

### H-2.4 Reagents — Authentic Samples (Purity Greater than 98 percent)

**H-2.4.1** 2-*mercaptobenzothiazo*le (MBT)

**H-2.4.2** *2,6 bis (1,1-dimethylethyl)-4- methyl-phenol* (Antioxidant BHT)

**H-2.4.3** *2,2'-methylenebis* [*6-(1,1-demethylethyl*]*-4-methyl-phenol)* (Antioxidant 2246)

### H-2.5 Reagents - Standard Solutions

**H-2.5.1** *Standard MBT Solution* — Prepare six standard solution containing for example 1.0 mg, 2.0 mg, 5.0mg 10.0 mg, 15.0 mg and 20.0 mg MBT/litre of acetonitrile.

**H-2.5.2** *Standard Antioxidants Solution* — Prepare a solution of the two antioxidants containing 30 µg Antioxidant BHT and 15 µg of antioxidant 2246 in 5 ml of acetonitrile .

### H-2.6 Procedure

Weigh 1 dm² or, if 1 dm² is not available ,the largest possible area of the pre-treated sample and cut it into as few parts as possible. The number of parts shall be defined by the size of the neck of a 250 ml flask. The area of the sample shall be the sum of the areas of the inner and outer surfaces.

Store the sample for 24 h in the aqueous migration liquids (water to represent milk and 3 percent acetic acid to represent fruit juices) at 40ºC in a drying oven in the ratio of 1cm$^2$ /2ml aqueous migration liquid. After removing the solid parts, shake the aqueous migration liquid with two 50 ml aliquots of dichloromethane. The combined organic phases are dried over anhydrous sodium sulphate and evaporated carefully to dryness. The residue is then re-dissolved in 5 ml of acetonitrile.

NOTES

**1** Cutting into two pieces is usually sufficient for a feeding teat.

**2** To aid measurement of area, cut the elastomeric or thermoplastic part into several pieces and draw around them on millimetre paper. Count the number of squares within each line and add the number together.

**3** Concentration columns may be used to replace shaking with dichloromethane.

### H-2.7 Calculation

**H-2.7.1** *MBT*

Inject the six standard solutions (*see* **L-2.5.1**) into a HPLC with HPLC column three times each. Produce a calibration curve of mg MBT/kg material using the eighteen values.

Inject the test solution (*see* **L-2.6**) into the HPLC .Use the calibration curve to determine the MBT – content of the test solution, either manually or with data-handling software. A detection limit of < 0.1 µg MBT/ml sample solution shall be obtained.

NOTES

**1** A suitable HPLC apparatus and method are described in Annex I.

**2** The calibration curve should be rectilinear and the correlation coefficient 0.997 or better.

**3** It is recommended that the test be carried out at least in duplicate.

**H-2.7.2** *Antioxidants*

Inject the standard solution (*see* **L-2.5.2**) into a HPLC with HPLC column. Inject the sample solution (*see* **L-2.6**) in the same way. Determine the amounts of migrated antioxidants ,in mg antioxidant/cm² material, by comparison of the peak areas in the chromatograms of the standard solution and the sample solution either manually or with data handling software.

If the peak areas of the antioxidants in the test solution are greater than the standard peak areas, prepare and obtain chromatograms of additional standard solutions in order to create a calibration curve over the region of interest. Obtain the amounts of migrated antioxidants from the calibration curve.

NOTES

**1** A suitable HPLC apparatus and method are described in Annex I.

**2** It is recommended that the test be carried out at least in duplicate.

# ANNEX J

(*Informative, Clauses* 5.3.5 and 5.3.6)

## SUITABLE HPLC APPARATUS AND METHOD FOR THE DETERMINATION OF 2-MERCAPTOBENZOTHIAZOLE (MBT) AND/OR ANTIOXIDANTS

**J-1** The following column has been found to be suitable:

Reversed phase $C_8$, for example Spherisorb $C_8$.

5 µm diameter, length 25 cm.

**J-2** The following operating conditions have been found to be suitable for this column.

**J-2.1** Mobile phase (Eluent A) — water containing 1percent acetonitrile, and

**J-2.2** Mobile phase (Eluent B) — acetonitrile.

The mobile phase may require degassing.

**J-3** The gradient programme is shown in Table 5:

**Table 5 Gradient Programme**
(*Clause* I-3)

| Sl No. | Time (min) | Percent Eluent A | Percent Eluent B |
|---|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | 0 to 2 | 70 | 30 |
| ii) | 2 to 17 linearly to | 10 | 90 |
| iii) | 17 to 22 | 10 | 90 |
| iv) | 22 to 25 linearly to | 70 | 30 |
| v) | 25 to 28[1)] | 70 | 30 |

[1)] Or longer, if further equilibration is thought to be necessary

The gradient of the eluent may need to be adjusted, if a different column to that described above is used.

**J-4 FLOW RATE** — 1 ml/min.

**J-5 DETECTION**

a) MBT — UV 320 nm, Diode array spectrum from 240 nm to 360 nm. Detector programming from time 5 min to time 12 min.

b) Antioxidants — UV 280 nm, Diode array spectrum from 240 nm to 360 nm. Detector programming from time 12 min to time 25 min.

**J-6 RETENTION TIMES**

a) MBT — approximately 9 min (*Max* 320 nm);

b) Antioxidant BHT — approximately 20 min (*Max* 278 nm);

Antioxidant 2246: approximately 21 min (*Max* 282 nm)

**J-7 INJECTION VOLUME** — 20 µl.

**J-8** Depending on the type of equipment used, the appropriate operating conditions may need to be established.

**J-9** Typical chromatograms for MBT and the antioxidants BHT and antioxidant 2246 are shown in Fig. 2.

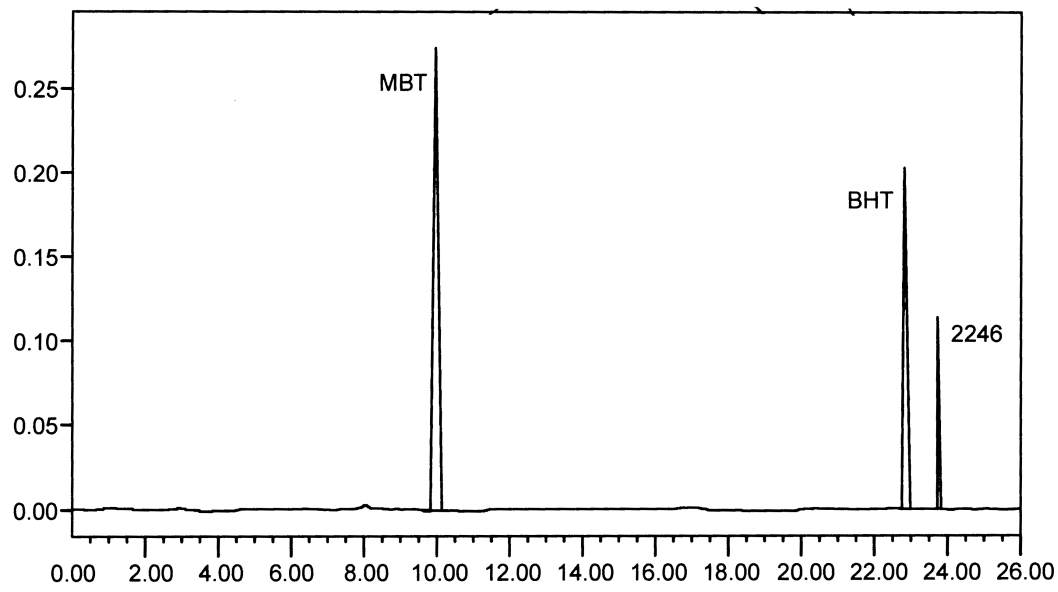



FIG. 2 TYPICAL CHROMATOGRAMS FOR MBT AND THE ANTIOXIDANTS BHT AND 2246
{ABSORBANCE (ABSORBANCE UNITS) VERSUS. RETENTION TIME (*Min*)}

# ANNEX K

(*Clause* 5.4.2)

## TEAR RESISTANCE TEST

### K-1 PROCEDURE

Place the teat on a cutting board of at least 10 mm thickness and (70 ± 5) Shore D hardness (equivalent to 97 IRHDs) as shown in the Fig. 3. Place the tip of the indentor (made of material of high chrome tool steel or equivalent, harden to 45-50 Rockwell C) centered over, and at right angles to, the major axis of the teat, in the region of the waist or neck of the nipple of the teat (that is 15 mm to 20 mm) from the tip of the nipple (*see* Fig. 4).

In the case of a feeding teat not having a circular cross-section, the indenter shall be placed over the flattened surfaces of the neck of the teat.

At a cross head speed of (10 ± 5) mm/min apply a force of 200 ± 10 N for 1 ± 0.5 s (*see* Fig. 3).

If the indentor punctures the component, teats may be subjected to tensile test.

NOTE — Before use, the tip of the indenter should be visually inspected. If any change is observed, the indenter should not be used as the results of the test may be affected.

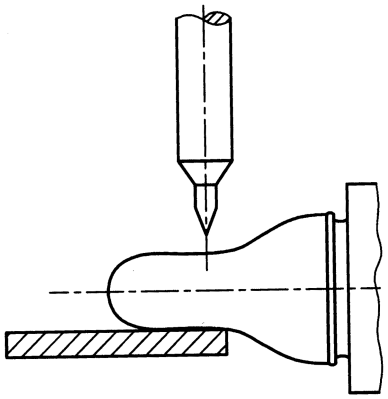


FIG. 3 POSITION OF TEAT FOR TEAR RESISTANCE

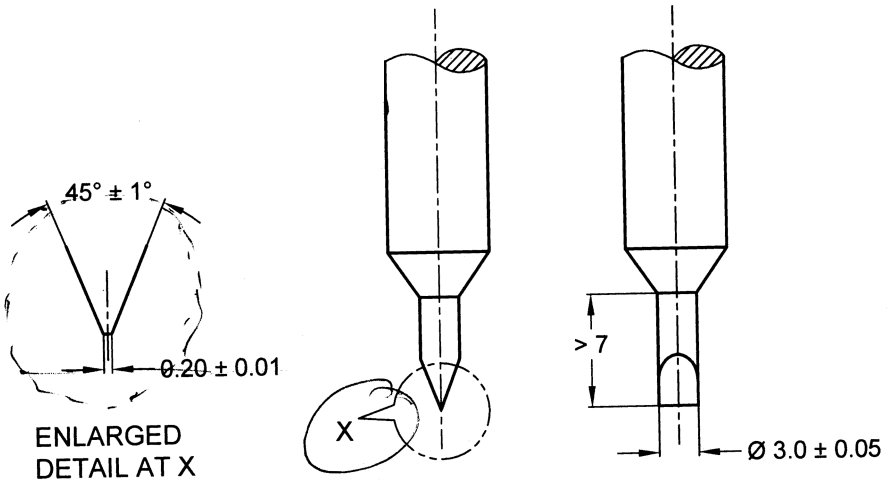



FIG. 4 SAMPLE INDENTOR

# ANNEX L

(*Clause* 5.4.3)

## TENSILE TEST

### L-1 PROCEDURE

A suitable fixing device shall be used to hold opposite ends of the feeding teat securely along the major axis. Apply a force of 5 ± 2 N along the major axis to align the specimen before increasing the force to 90 ± 5 N for 10 ± 0.5 sec at a crosshead speed of 200 ± 10 mm/min. Maintain for 10 ± 0.5 s.

Clamps or other devices shall hold the components securely during the test without causing which might affect the test result.

The sample shall be taken to have pass the test if no teat sample gets torn during the test.

# ANNEX M

(*Clause* 7)

## SAMPLING OF TEATS FOR FEEDING BOTTLES

### M-1 GENERAL REQUIREMENTS

Precautions shall be taken to protect the samples, the material being sampled and the containers for samples from adventitious contamination.

The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material of the teats has no action. The sample containers shall be of such size that they are almost completely filled by the sample.

Each sample containers shall be sealed air-tight and marked with details for identification like name of manufacturer, lot number, lot size, month and year of manufacture and date of sample collection.

### M-2 SCALE OF SAMPLING

#### M-2.1 Lot

All the rubber teats for feeding bottles of the same size and manufactured from the same raw materials under similar conditions of manufacture in one consignment shall constitute a lot.

Samples shall be tested from each lot separately, for ascertaining conformity of a lot to the requirements of this specification.

The number of teats to be selected in the sample from a lot shall depend upon the size of the lot and shall be in accordance with Table 6.

A representative sample shall be collected from each lot. At least 10 percent of the packages shall be selected at random. Equal number teats may be randomly drawn, from each selected package to give the required number of pieces as stated in col 2 of Table 6.

### M-3 CRITERIA FOR CONFORMITY

A lot of teats shall be conforming to the requirements of the standard, if the following are satisfied.

**M-3.1** Number of teats selected and tested as per col 2 of Table 6, shall not have defective teats exceeding the number given in col 3 of Table 6.

**M-3.2** All the tests results for various characteristics shall satisfy the requirements of the specification individually.

**Table 6 Number of Teats to be Selected from a Lot and Permissible Number of Defectives**

(*Clause* L-2.1)

| Sl No. | No. of Teats in the Lot | No. of Teats to be Selected in the Samples | Permissible No. of Defective Teats for Workmanship and Finish | No. of Times Each Test has to be Performed for Other Characteristics |
|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (5) |
| i) | Up to 3 000 | 90 | 2 | 1 |
| ii) | 3 001-10 000 | 180 | 4 | 2 |
| iii) | 10 001-35 000 | 270 | 6 | 3 |
| iv) | 35 001 and above | 450 | 8 | 5 |

NOTE — Each test shall be carried out on randomly selected test piece (s), collected as per column 3-6.

# ANNEX N

(*Informative*)

## INFORMATION ABOUT SILICONE RUBBER

### N-1 DEFINITION

Silicones constitute a group of polymeric chemical substances and preparations, all containing polysiloxanes. Polysiloxanes are characterized by Si-O-Si and Si-C bonds. They are prepared from chlorosilanes which are subjected to hydrolysis. Polysiloxanes such as fillers, emulsifiers and are also covered by the definition.

Furthermore, copolymers or polymer-blends of polysiloxanes with organic polymers are covered by the term 'silicones', provided siloxane monomer units predominate by weight over each of the other monomer units present. Silicones used as food additives are not covered by the definition.

### N-2 DESCRIPTION OF THE SILICONE PRODUCT GROUP

Silicones include a range of products with a variety of properties and application:

**N-2.1 Silicone Elastomers**: Coatings, sealants, etc.;

**N-2.2 Silicone Liquid**: Additives to plastics and coatings, release agents for moulding plastic articles, impregnating agents for textiles, etc.;

**N-2.3 Silicone Pastes**: Lubricants for food processing machinery, etc;

**N-2.4 Silicone Resins**: Heat-resistant coatings, release coating in food production such as bakery, etc.

### N-3 SPECIFICATIONS

Silicones used for food contact applications, hereafter called silicones, should meet the following requirement:

**N-3.1** They should not transfer their constituents to foodstuffs in quantities which could endanger human health or being about an unacceptable change in the composition of the foodstuffs or deterioration in the organoleptic characteristics thereof;

**N-3.2** They should be manufactured in accordance with a certified Quality Assurance System (for example ISO 9002 or CEN 29-004) and under the conditions specified in 'Technical document No. 1 – List of substances used in the manufacture of silicones used for food contact applications'.

**N-3.3** Interactions between starting substances or between starting substances and any other substance used in the manufacturing process of silicones should not lead to the formulation of compounds which pose a risk to human health;

**N-3.4** linear siloxanes containing methyl-and phenyl-groups attached to the same silicone atom and also containing two methyl-groups attached to one silicon atom (siloxanes and silicones, Me-, Ph-, di-Me-) should not be used as starting substances, in order to prevent the formulation of certain phenyl-substituted cyclic polydimethylsiloxanes;

**N-3.5** The release of any substance from silicones to foodstuffs should be as low as technologically possible. The total of all substances migrating into food from silicone materials or articles should not exceed 10 mg/$dm^2$ of the surface area of the final material or article or 60 mg/kg of food, this being considered as the overall migration limit;

**N-3.6** The migration limits set out in this standard;

**N-3.7** Migration tests should be conducted according to Directives 82/711/EEC, 90/128/EEC, 93/8/EEC, 97/48/EEC and their future amendments, as appropriate, unless technically impracticable due to the nature of the material and the migration test;

**N-3.8** Appropriate labelling for the materials or articles should be provided, if pre-washing by the user is necessary.

# ANNEX P

(*Foreword*)

## COMMITTEE COMPOSITION

Rubber and Rubber Products Sectional Committee, PCD 13

| *Organization* | *Representative(s)* |
|---|---|
| Rubber Board, Kottayam | Shri N. Rajagopal (***Chairman***) |
| All India Rubber Industries Association, Mumbai | Shri D. J. Bharucha<br>Shri Amlesh Roy (*Alternate*) |
| Apollo Tyres Ltd, Kerala | Dr Arup K. Chandra<br>Shri T. D. Varkey (*Alternate*) |
| Association of Planters of Kerala, Thirupuram | Shri A. Jacob |
| Automotive Tyres Manufacturers Association (ATMA), New Delhi | Shri Vinay Vijayvargia<br>Shri Vijay Sethi (*Alternate*) |
| Central Revenues Control Laboratory (Ministry of Finance, Department of Revenue, Central Board of Excise & Customs), New Delhi | Shri Suneel Mathur |
| Dow Corning India Pvt Ltd, Mumbai | Shri Subhranshu Gupta |
| Export Inspection Council of India, Ministry of Commerce & Industry, New Delhi | Shri K. J. Srivastava<br>Shri S. K. Saxena (*Alternate*) |
| Flame Retardants Association of India, Gurugram | Shri Murali Mohan |
| GRP Limited, Mumbai | Shri Mehul Patel<br>Shri Kalyan Das (*Alternate*) |
| HASETRI, Rajasamand, Rajasthan | Dr Saikat Das Gupta |
| HLL Lifecare Limited, Trivandrum | Shri H. U. Kantharaju |
| Indian Rubber Manufacturers Research Association, Thane | Shri K. Rajkumar |
| KA-prevulcanized Latex Pvt Ltd, Nagercoil | Dr R. K. Matthan<br>Shri Joseph John (*Alternate*) |
| LPG Equipment Research Centre, Bangaluru | Shri P. Krishnan Kutty<br>Shri R. Rajkumar (*Alternate*) |
| Ministry of Defence (DGQA), New Delhi | Shri S. K. Saxena<br>Shri V. K. Chhabra (*Alternate*) |
| Phoenix Yule (P) Ltd, Kolkata | Shri Soumitra Ganguli<br>Shri Asok Kumar Ghosh (*Alternate*) |
| Reliance Industries Ltd (Elastomers Business), Vadodara | Shri Bharat B. Sharma |
| Rubber Board, Kottayam | Dr Siby Varghese |
| Research, Designs & Standards Organization, Lucknow | Shri Suraj Singh |
| Voluntary Organization in Interest of Consumer Education (VOICE), New Delhi | Shri M. A. U. Khan<br>Shri H. Wadhwa (*Alternate*) |
| BIS Directorate General | Shri A. K. Bhatnagar, Scientist 'F' and Head (PCD)<br>[Representing Director General (*Ex-officio*] |

*Member Secretary*
Shri Chandrakesh Singh
Scientist 'C' (BIS)

(*Continued from second cover*)

responsibility of the manufacturer to ensure appropriate health and safety precautions are taken in its manufacture and determine the applicability of the regulatory requirements related to the product.

The composition of the Committee, responsible for the formulation of this standard is given at Annex P.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 13 (10468).

**Amendments Issued Since Publication**

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
| | | |
| | | |
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